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ON PACHNOLITE AND THOMSENOLITE.

BY GEORGE AUG. KOENIG, PH.D.

In a very able paper ("Ann. Chem. & Pharm.," vol. cxxvii. 61, 1863), A. Knop called the attention of mineralogists to two forms of a mineral, which occurs incrustating the cryolite from Arksudfiord, Greenland. One kind he describes as rectangular parallelepipedic crystals, which are possessed of three perfect, but unequal, cleavage directions, parallel to the faces of the crystal, the latter being mostly covered with ferric hydrate. The cleavage directions seemed to coincide with those of the underlying cryolite (identified by quantitative analysis); but an accurate determination of the angles was not possible on account of insufficient reflecting power of the faces. Approximately they were found to be 90° .

The second form of crystals occurs in cavities, whose walls are covered with brilliant, colorless, and transparent crystals.

Both kinds were found chemically identical, and, therefore, belong to one mineral, to which Knop gave the very characteristic name, *Pachnolite*—frost stone—from the frost needle-like incrustations covering the cryolite.

From measurements of the small crystals, they were found to belong to the rhombic system, offering combinations of $\infty P.P$; $\infty P.oP.P$; and $\infty P.oP$. The first kind of crystals are of the combination, probably, $oP.\infty\bar{P}\infty.\infty\bar{P}\infty$. The small crystals show a perfect basal cleavage. The angles were found $\infty P : \infty P = 81^\circ 24' - 98^\circ 36'$ (mean of 12 determinations). $P : \infty P = 154^\circ 40'$ (mean of 5 measurements).

Other angles were deduced by calculation:—

Specific gravity = 2.923.

Composition found in mean

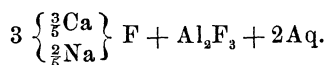
F	= 50.79 : 19	= 2.673	6.202
Al	= 13.14 : 27.5	= 0.477	1.106
Na	= 12.16 : 23	= 0.530	1.206
Ca	= 17.25 : 40	= 0.431	1.000
HO	= 9.60 : 18	= 0.533	1.236

102.94

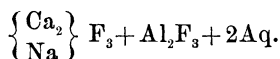
Knop takes the atom $\text{Al} = 13.75$, and $\text{Ca} = 20$, $\text{HO} = 9$, therefore his ratio is

F	Al	Na	Ca	HO
6.20	2.21	1.20	2.00	2.236

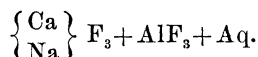
and he constructs the formula



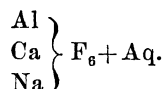
This, however, is not a correct deduction, because $\text{Ca} : \text{Na}$ is not $3 : 2$, but much nearer $2 : 1$, and the formula must necessarily be



Or, if we introduce the present atomic weights, the formula is



or the empirical formula



It will be seen from the foregoing that, taking the calcium as unit, all the other atoms are too high, and that the analysis adds up to nearly 103.00, which is rather more than the mean from several analyses should be. Considering, however, the nature of the substance, too high a result is explicable, and the formula as deduced by me, being of the greatest simplicity at the same time, may be taken to represent fairly the molecule of *Pachnolite*.

It is to be regretted that Knop does not state whether he used the small brilliant crystals affording the above crystallographic results, or whether he used the larger parallelepipedic crystals, or both. It should never be omitted to describe exactly the material taken for analysis, and how it was selected.

Knop's erroneous formula is admitted into Dana's "Handbook" without challenge.

Hagemann also published an analysis of *Pachnolite* ("Am. Journ." ii. xli. 119), which yields the following atomic ratio:—

F	Al	Na	Ca	H ₂ O
6.17	0.865	1.200	1.00	1.09.

Ca : Na is nearer 2 : 1 than 3 : 2 (taking Ca = 20) as it is in Knop's analysis, and confirms the above formula.

Professor Dana, in the fifth edition of his "Handbook of Mineralogy," introduces a new species, *Thomsenolite*, which had been described by Hagemann ("Am. Journ. Sci." ii. xlii. 93) as *Dimetric Pachnolite*.

Hagemann made no crystallographic determinations, except what may be adduced with the naked eye, and his description coincides with that given by Knop for the variety A of *Pachnolite*, the parallelepipedic crystals of the combination $\infty \bar{P} \infty, \infty \bar{P} \infty .oP$.

According to Dana, the crystals are monoclinic prisms 89° — 91° — $0^\circ 1' = 92^\circ$ and $88''$.

The faces of the prism are usually striated horizontally, cleavage basal, very perfect.

Specific gravity, 2.74–2.76. Lustre vitreous, of a cleavage face a little pearly, color white or with a reddish tinge.

Composition.	Na = 1, the ratio is
F = 50.08 : 19	= 2.63
Al = 14.27 : 27.5	= 0.515
Ca = 14.51 : 40	= 0.362
Na = 7.15 : 23	= 0.311
H ₂ O = 9.70 : 18	= 0.54

Taking Na = 0.311 as unit, the atomic ratio becomes

F	Al	Ca	Na	H ₂ O
8.46	1.656	1.109	1.000	1.740

and reducing to whole atoms, the nearest approach is

$$17 : 3 : 2 : 2 : 3$$

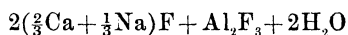
or

$$\left. \begin{array}{l} Al_3 \\ Ca_2 \\ Na_2 \end{array} \right\} F_{17} + 3Aq.$$

This formula appears improbable from its complexity, and from the fact that the affinities of fluorine are not satisfied by the metals. Dr. Hagemann states the crystals to have been covered with a white earthy material (SiO₂?), and accounts for SiO₂ = 2.00 in the analysis.

Whenever a mineral substance is so obviously heterogenous as this one, its analysis should not be considered reliable enough to deduce a formula, or form an opinion of molecular composition.

Dr. Hagemann's formula



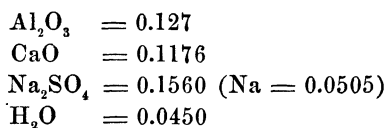
is not consistent with the analysis; it is a mere conjecture, as may be seen by comparing with the above atomic ratio.

Having lately obtained a number of specimens from the Greenland cryolite locality, through the Reverend Dr. Beadle, of this city, to whom I herewith express my thanks, I thought it worth the trouble to corroborate the few analyses of these very interesting minerals.

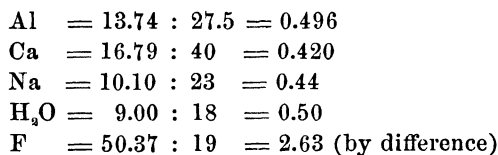
A specimen, agreeing completely in its physical properties with the description of Knop's variety A of pachnolite, was first investigated. The structure of the specimen is very like that of crusts of salt, as they are often obtained by slow evaporation—tabular aggregations of cubes, arranged parallel to each other, and at right angles, leaving interstices between themselves into which the cubic crystals project. The tabular masses have apparently one common cleavage face for all individuals, which is of a decided pearly lustre, as described for Thomsenolite. The faces projecting into the interstices are striated and tapering. The crystals are perfectly colorless for the most part. The basal plane O is well developed in all individuals, but the pyramidal faces 1, 1, are usually suppressed. Some of the projecting prisms carry very small octahedrons, either Chiolite or Ralstonite. In selecting the material for analysis, the greatest care was taken to select only perfectly clear cleavage crystals, on which any admixture might be most easily discovered.

An attempt to measure the angles of cleavage direction proved unsatisfactory; the measurements differed several degrees, but (in most cases) were found to be near 90°.

Spec. grav. = 2.937 (made with 5.6921 grammes); 0.5000 grm. gave—



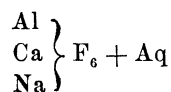
1.0000 grm. gave NaCl = 0.2535 (Na = 0.0997) and MgO = 0.0023.



Taking Ca = 0.42 as unit, the ratio is—

F	Al	Ca	Na	H ₂ O
6.30	1.17	1.00	1.05	1.30

agreeing to the formula—



as deducted from Knop's analysis of Pachnolite.

I selected now another specimen, which contains very brilliant crystals in a druse. I broke the crystals off with a forceps, so as to leave a stump on the matrix to be sure of a thoroughly homogeneous material.

These crystals were very slender, of quadratic section, and gently tapering to a point. The basal plane O seemed entirely suppressed in nearly all the crystals, and the pyramidal faces in many, but the very brilliant faces of the prism were distinctly striated horizontally. Basal cleavage very perfect, with pearly lustre. A series of measurements with specimens about $\frac{1}{8}$ inch long and $\frac{1}{32}$ inch wide gave for the prismatic angles the following figures:—

1	2	3	4
90° 30'	90° 10'	90° 15'	90° 5'
89° 36'	89° 52'	89° 25'	90° 15'
90° 14'	90° 13'	90° 15'	89° 25'
89° 40'	89° 45'	90° 5'	90° 15'

The angles are not very constant, but the deviation from a right angle is very small. The angle of the basal plane with the prism could not be determined to my satisfaction. Considering the tapering forms, it seems impossible to say whether the form is rhombic or monoclinic, or quadratic. The points of all the crystals were colored yellow or brown by ferric hydrate, and some crystals had a light straw-color all through.

Specific gravity = 3.008 (determined with 0.7153 grm. in a pyknometer holding about 2 cub. cent. of water).

0.5000 grm. gave—

Al ₂ O ₃	= 0.1170
CaO	= 0.1270
Na ₂ SO ₄	= 0.1575 (Na = 0.0511)
H ₂ O	= 0.0252 (from 0.3075 grm.)

Yielding percentage—

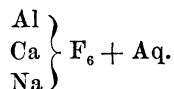
Al	=	12.50 : 27.5	=	0.454
Ca	=	18.14 : 40	=	0.453
Na	=	10.23 : 23	=	0.444
H ₂ O	=	8.19 : 18	=	0.455
F	=	51.54 : 19	=	2.702

100.60

Taking Na = 0.444 as unit, the ratio obtains—

F	Al	Ca	Na	H ₂ O
6.080	1.042	1.020	1.000	1.030

or—



(The fluorine is calculated for the percentages of the metals.)

About the correctness of this formula, and the true molecular composition of the mineral, there can be no longer any doubt in view of the above analytic results.

A mineral occurring in small stalactitic and warty masses which project from parallel walls or partitions made up of a compact mineral. It is colored strongly brown, and shows a velvety lustre. On closer observation the stalactites and warts appear to be aggregations of very minute prismatic needles of strong vitreous lustre. No selection was attempted.

0.5000 grm. gave—

Al ₂ O ₃	=	0.1235
CaO	=	0.1195
Na ₂ SO ₄	=	0.1135 (Na = 0.0367)
H ₂ O	=	0.047
F ₂ Ca	=	1.0577 (F = 0.5194) from 1.000 grm.

Yielding percentage—

F	=	51.94 : 19	=	2.734
Al	=	13.16 : 27.5	=	0.478
Ca	=	17.07 : 40	=	0.429
Na	=	7.35 : 23	=	0.320
H ₂ O	=	9.40 : 18	=	0.525

98.92

Taking Ca = .429 as unit, the ratio obtains—

F	Al	Ca	Na	H ₂ O
6.3	1.11	1.00	0.746	1.22

The sodium is too low in this ratio to admit of anything more than an approximation to the general formula, and I consider this substance as a mixture like the one analyzed by Dr. Hagemann (*l. c.*).

The blowpipe and other general chemical properties I found to be as stated by Knop. In analyzing these substances it is necessary to evaporate the solution in the sulphuric hydrate to dryness, to redissolve by boiling with about 300 cubic centimetres of water slightly acidulated, when all the calcium sulphate will pass into solution.

In separating calcium and aluminum I encountered no difficulty, although the aluminum hydrate is very gelatinous. Filtering under pressure, without washing the precipitate, I found, after redissolving it in HCl and reprecipitating by NH₄HO, but a trace of Ca in the filtrate.

It is necessary, however, to ignite the aluminum oxide on a blast in order to obtain a correct weight. In decomposing the mineral in HCl it dissolves, as in H₂SO₄, to a viscous mass, but a complete elimination of fluorine was not effected even after evaporating with strong acid (to dryness 6 times). The aluminum precipitate contained about one-half of the calcium as fluorid.

As is well known, the determination of fluorine presents a number of difficulties, which render an accurate result very uncertain. Fresenius's method, although capable of yielding reliable results, is nevertheless almost impracticable, from the accumulation of errors by changing weights in the numerous parts of desiccating and absorbing apparatus. I endeavored, therefore, to set the fluorine free as fluorid of hydrogen, and collect the latter in an alkaline solution. This method has been proposed (Rose—Analytical Chem.), but I am not aware whether it was ever practised with natural fluorids. Having a platinum still at my disposal, I thought of testing the applicability to the analysis of the above minerals.

In a first experiment sulphuric hydrate was used to decompose the mineral at a temperature rising gradually to the boiling point of the acid. After 45 minutes, on opening the still and dropping in

water, a strong disengagement of HF took place. In a second experiment one part of hydrate was mixed with one part of water. The distillation proceeded very well, and was only interrupted after the alkaline liquid in the receiver (containing 25 p. c. more of NaHO than was approximately required by the fluorine) turned acid. On opening the still and adding water, no HF was disengaged, and the entire residue from distillation passed into solution when heated with a large quantity of water; hence a complete decomposition of the mineral had taken place, and the acid vapors had carried all HF into the receiver. After neutralizing the liquid in the latter, a solution of CaCl_2 was added, containing slightly more than the fluorine would prospectively require, and the liquid heated to boiling. The calcium fluorid coagulated perfectly and filtered very easily. It was twice returned into the capsule and boiled with water to extract all calcium sulphate. The precipitate weighed after ignition 1.0577 grammes.

To the filtrate a solution of sodium carbonate was added in excess and boiled, the precipitate ignited, and extracted with precaution by acetic acid. It weighed 0.0085 after being again ignited. The alkaline filtrate was acidulated and precipitated by BaCl_2 . Precipitate weighed 0.1580. In order to ascertain the exact quantity of sodium hydrate which had been combined with the acids, an equal volume (30.2 c.c.) was evaporated with HCl. The sodium chlorid weighed 1.7205 grms. = 0.9117 Na_2O . But 0.2 c.c. had been used to restore the blue color, and has to be subtracted, giving $0.9117 - 0.0060 = 0.9057 \text{ Na}_2\text{O}$ combined with fluorine and sulphuric anhydride.

We found—

BaSO_4 0.1580 = SO_3 — 0.0542, requiring

0.0420 Na_2O , hence

$0.9057 - 0.0420 = 0.8637 \text{ Na}_2\text{O}$ was combined with fluorine, but
 $31 \text{ Na}_2\text{O} : 19 \text{ F} = 0.8637 : 0.5230$.

By precipitation was obtained 0.5194 F, a difference of 0.0036.

This result was quite promising. It was obtained with the stalactitic aggregations of pachnolite. But on applying the method to the analysis of the parallelopipedic crystals of pachnolite, I encountered difficulties quite unaccountable. I did not succeed in decomposing the mineral completely, either with one part of sulphuric hydrate and one part of water, or with more dilute acid, or by fusing with KHSO_4 in repeated trials and proportions.

I am, however, still confident that the method can be so modified as to be applicable to these fluorids.

Regarding silicium dioxide, which Hagemann found in his analysis, I endeavored to find it, but failed. It was certainly owing to superficial impurity.

Potassium I could separate in traces only.

The determination of water I found to be most satisfactory when I used calcium oxide mixed with the minerals instead of lead oxide. The latter when heated to expel moisture is very apt to be partially converted into sesquioxide, which will at a red heat lose oxygen, and the quantity of water will be found too high.

Conclusions. 1. The mineral analyzed by me is identical in composition with Knop's pachnolite.

2. It is identical in form and physical properties with thomsonolite.

3. The measurements are so uncertain that the true form of the parallelopipedic crystals cannot be deduced, and the form may be explained as Knop did.

4. The mineral measured by Knop and Descloizeau has *perhaps* not been analyzed, since Knop does not describe his material taken for the analysis.

5. From the foregoing it does not seem justified to separate the parallelopipedic forms as a distinct species, and the name pachnolite being very expressive and older, all the forms should be designated as *pachnolite* until further investigation.

I may be permitted to state that I do not intend to discard the crystallographic results of Knop, Dana, and Descloizeaux (the original of the latter's work I am unacquainted with). I should be very glad to obtain the crystals which gave the prismatic angles 98° and 81° , so that we should know whether there are two different molecules with two different forms, or whether there is only one dimorphic molecule. But from the measurements I was able to make, I should be very doubtful of dimorphism.

Note.—While this paper is in press, I find a very recent publication by Professor Wöhler (*Ann. d. Chem. u. Pharm.* vol. clxxx. p. 231), in which he gives an analysis and description of the variety A of pachnolite, and arrives at results closely corresponding to my own.